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THE CARBON SUBSULFIDE C_3S_2

by

A. Stock
P. Praetorius

Chemische Berichte, 45, 3568-3578 (1912)

Translated from the German

March 1967

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A more thorough study of the recently described[1] carbon telluride (produced through evaporation by tellurium electrodes, using an arc light in the presence of carbon disulfide) showed that it was always mixed with a sharp smelling substance* formed of sulfur and carbon. This proved to be the compound C_3S_2, described by von Lengyel 20 years ago in a little-noticed article[2]. Von Lengyel designated it as "tricarbonium-disulfide". Since in all respects — formula, external characteristics, ease of polymerization, and also constitution — it corresponds (as we shall show here) to the carbon suboxide, C_3O_2, discovered by Diels and Wolf, it is called for practical purposes "carbon subsulfide". Von Lengyel obtained carbon subsulfide by letting an arc light burn for a few hours between carbon electrodes in the vapors of carbon disulfide (boiling and connected to a reflux condenser). In addition to solid carboniferous products, a red solution was formed which was treated for several days with copper shavings to remove the sulfur. Evaporation in a dry air current produced a red liquid with an unbearably sharp smell, able to be distilled almost undisturbed in a vacuum at 60°-70°, and having a very low carbon disulfide content. This liquid was characterized by von Lengyel as C_3S_2, through fairly valid analyses and by determination of the molecular weight (performed by the freezing point method with benzene as solvent). He further reported that, when combined with bromine, C_3S_2 can be transformed into a stable, solid, yellow bromide, $C_3S_2Br_6$; that in free form it converts very quickly into black substances, probably polymerization products; and he gave some other, qualitative data about the new material, whose constitution he discussed, but did not specify.

For the continuation of our work on carbon telluride we needed a more exact knowledge of carbon subsulfide. On this occasion we produced it in an entirely pure state; tested different ways of producing it; specified certain constants of the pure preparation; and were able to prove, through the smooth transition of C_3S_2 combined with aniline into the already familiar compound thiomalonic anilide, that it is to be understood as S:C:C:C:S, an anhydride of thiomalonic acid.

C_3S_2 can be produced from carbon disulfide by various methods. Dilute carbon disulfide solutions are always obtained, in which in addition to C_3S_2, changing quantities of impurities, such as sulfur and perhaps also carbon polysulfides, are to be found. These impurities — which remain behind when the solution is evaporated, while C_3S_2 evaporates — can be removed by treating the liquid with

*Perhaps the penetrating odor of carbon telluride is due only to this impurity.

copper, and also by the more effective and much quicker method of shaking with mercury*, already used by Lengyel. We determined the carbon subsulfide content of the remaining pure C_3S_2 solutions by evaporation with excess bormine and weighing of the residue, whose composition corresponds to the formula $C_3S_2Br_6$ (found: 82.0 percent Br, 11.3 percent S; calculated: 82.7 percent Br, 11.1 percent S). We satisfied ourselves time and again that the conversion of C_3S_2 into the bormide proceeds quantitatively.

The production of the C_3S_2 solution according to von Lengyel is not a very pleasant operation. We used the apparatus described and pictured in von Lengyel's report. Since measurements were not given, we made the size correspond as nearly as possible to the drawing (400 ccm, flask over it a spherical continuation with a capacity of about 1 liter for the arc light).

The glass vessel, which is greatly heated by the arc light then cooled in spots by the carbon disulfide flowing back from the condenser, soon broke. The yield of C_3S_2, after the arc light had been burning for 15 minutes (30 amperes, 12 mm thick electrodes), was 0.05 g to 0.09 g, according to whether a shorter or longer arc light was used. The quantity of the products formed besides C_3S_2 (hereinafter designated simply as "residue") had a ratio of 1.6:1 to that of carbon subsulfide; if the carbon disulfide simply decomposed during the reaction:

$$3CS_2 = C_3S_2 + 4\,S$$

(whereby the residue should be pure sulfur, which, however, is not the case), this ratio would then be 1.3:1. Von Lengyel does not give his yields of pure C_3S_2. After burning the arc (10-40 amperes) for 2-3 hours and blowing air over the solution (which had been filtered and treated with copper), he obtained 2-3 g of a liquid, which, however, must still have contained much carbon disulfide.

When we allowed an arc light to burn between 6 mm thick graphite electrodes in liquid carbon disulfide (110 volts with resistance; 15 amperes; 350 ccm CS_2), after a burning time of 28 hours 1.25 g of C_3S_2, in addition to many by-products (solid black particles; dark brown solution), were formed.

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*The copper sulfide formed from the copper also decreases the C_3S_2 content of the solutions rather quickly (see below). Mercury causes noticeable losses only when the quantity of impurities exceeds by far that of C_3S_2.

That the formation of C_3S_2 in these experiments is probably due to the dissociation of CS_2 by the heat and not to reduction by the carbon of the electrodes is shown by the fact that C_3S_2 also forms when CS_2 vapors are heated in a silica tube. We used an apparatus similar to that used in the already familiar production of diphenyl from benzene vapors. Purest dry carbon disulfide boiled in a flask in a water bath; its vapors streamed through an empty silica tube heated to 800°-1200°C and were condensed immediately thereafter in a condenser; the liquid flowed back into the flask. At the higher experimental temperatures in the heated portion of the silica tube black incrustations settled out; and in the colder portions farther back, sulfur, polymerization products of C_3S_2, etc. precipitated. At 800°C no decomposition of the carbon disulfide took place: the condensed liquid remained totally colorless*. Only toward 900°C did the formation of C_3S_2 begin. How the increase in temperature affected the quantities of C_3S_2 and of the "residue" is shown by the following table:

a) at 900°C in 12 hours 0.24 g C_3S_2 and 0.15 g "residue" (1:3.8)

b) at 1000°C in 12 hours 0.3 g C_3S_2 and 0.6 g "residue" (1:2.0)

c) at 1100°C in 12 hours 0.6 g C_3S_2 and 1.55 g "residue" (1:2.6)

d) at 1200°C in 12 hours 0.1 g C_3S_2 and 4.6 g "residue" (1:46)

As seen, the best yields of C_3S_2 were obtained at 1000°-1100°C. At a higher temperature more carbon disulfide is decomposed, it is true, but in another way. The carbon precipitated in the red-hot tube decreases the yield of C_3S_2. When we placed charcoal, carefully washed and first normalized in CS_2 vapor, in the silica tube, at temperatures between 900° and 1100°C we obtained no C_3S_2 at all, but in $2\frac{1}{2}$ hours over 5 g of "residue". In the same way the yield of C_3S_2 decreases when the experiment is continued with the silica tube (initially empty) and much carbon has been deposited in the tube. When we repeated Experiment b) with the silica tube already used and not yet cleaned, we obtained:

e) at 1000°C in 12 hours 0.24 g C_3S_2 and 0.7 g "residue" (1:2.9).

*Arctowski (see the article cited above) observed a partial decomposition of CS_2 when he distilled carbon disulfide 15 times in a combustion tube heated to 600°C. He evidently used impure carbon disulfide; for he also reports that carbon disulfide is greatly changed when heated in a closed tube at 175°C, which does not occur with pure material, even at 200°C.

The detrimental effect of carbon also explains why no C_3S_2 is found in industrial raw carbon disulfide.

In the presence of metals which can reduce carbon disulfide below the formation of sulfides, C_3S_2 forms at lower temperatures. When a silica tube filled with iron-wire spirals was used, the formation of very pure C_3S_2 could already be observed below 800 °C. It soon stopped, however, because the iron wire became coated with sulfide. In this way we got the idea of producing C_3S_2 by atomizing metals in liquid carbon disulfide with the arc light, a process in which constantly renewed layers of the reducing metal can act. We thereby returned to the procedure which had first brought us in contact with C_3S_2. The experimental set-up was the same for all following experiments.

In a water-cooled, 300 ccm flask were 200 ccm of carbon disulfide, containing two electrodes, one above the other, whose underneath parts were made of graphite and whose movable upper parts consisted of a 5-6 mm thick bar of the metal. A stream of carbon dioxide prevented the ignition of the carbon disulfide by the sparks springing occasionally from the electrodes. The length of the individual experiments, which were all begun with a current of 5 amperes (15 volt terminal voltage), varied according to the speed with which the respective metal evaporated. If this occurred easily, we used the metal as the cathode; otherwise, as the anode. The processing of the solutions, which here contained only minimal quantities of "residue", was done as described earlier.

Metal	Burning Time (min)	Metal Evaporated (g)	Metal Evaporated per hour (g)	C_3S_2 (mg)	C_3S_2 per g of Evaporated Metal (mg)	C_3S_2 per hour (mg)
a) Bi	15	12.0	48	7	0.6	28
b) Pb	25	12.5	30	12	1.0	29
c) Sn	18	7.0	23	18	2.6	60
d) Sb	25	7.0	17	16	2.3	38
e) Cd	60	3.5	3.5	21	6.0	21
f) Zn	60	0.8	0.8	32	40	32
g) Sb + 7%C	60	3.1	3.1	160	50	160

In all cases large quantities of metal sulfide were formed, which supports the fact that C_3S_2 was here produced by the reduction of carbon disulfide.

Copper, iron, and aluminum electrodes did not evaporate at all and produced almost no C_3S_2. Arsenic electrodes yielded abundant C_3S_2, but at the same time much yellow arsenic[3], which contaminated the solution.

Experiments a) to f) show that the C_3S_2 yield is significantly dependent on the electrode metal and allow the observation, among others, that relatively little C_3S_2 (with relation to the use of electrodes) forms when the respective metal evaporates very quickly. Accordingly, the C_3S_2 yields rose, even in the case of electrodes made from easily evaporated metals, when we artificially decreased their rate of evaporation. This was done by the addition at will of graphite powder*. For these experiments we used antimony, because it was easiest to pulverize and to mix with graphite. The most C_3S_2 was produced by electrodes of anitmony with about 7-percent graphite, as in Experiment g). The yield (easily raised by increasing the current) is in this case so high and attained in such a relatively short time, that the method presents itself as feasible for the practical production of carbon subsulfide and was exclusively used by us in the creation of our raw material for the experiments described later on. With a larger quantity of graphite, the electrodes burn down too slowly.

We obtained carbon subsulfide in the following way:

40 g of powdered antimony (Kahlbaum, A. G.)**, which has been sifted through silk, and 3 g of finest graphite powder (Acheson Graphite Co., Niagara Falls; "Polishing Lead" brand) are thoroughly mixed, kneaded with 4 ccm sugar solution (1:1), and pressed in a lightly oiled steel cylinder with a tightly fitting screw-actuated plunger to an electrode 12 mm thick and 6-8 cm long. We did this in a large lathe, leaving the electrode in the form for several hours and raising the pressure from time to time; most of the sugar solution is in this way squeezed out. The highly breakable electrode is pressed carefully out of the form, dried for 12 hours in a vacuum desiccator over phosphorous pentoxide, and then heated for a few hours time in a carbon dioxide stream to about 360°C. It is now

*Whether the graphite powder itself favors the formation of C_3S_2, and to what extent, cannot be determined at this time.

**The antimony must be free of arsenic, otherwise yellow arsenic will form later.

ready for use and fairly resistant. With a little practice, flawless electrodes are easily produced.

750 ccm of pure carbon disulfide (shaken with mercury and phosphorous pentoxide and distilled) are placed in a 1 liter round-bottom flask which is cooled by running water, including the neck. A stopper with three holes — for a carbon dioxide supply tube and for the approximately 6 mm thick brass suspension wires of the two electrodes — is set loosely into the neck. The cathode consists of a small graphite cylinder, about 1 cm high and at least $1\frac{1}{2}$ cm in diameter*; it is held on its underside by the brass wire, bent twice at right angles, close over the bottom of the flask. Above it is the antimony-graphite anode; it is held in a brass casing fastened to the end of the suspension wire. The wire glides with little friction through a brass tube in the stopper, so that the anode can be easily raised and lowered.

Power supply: 110 volts with resistance; Current: 10-15 amperes; Terminal voltage during the experiment: approximately 30 volts.

As carbon dioxide is slowly fed in, the two electrodes are brought into contact and then drawn a little apart, so that an arc light is formed. The liquid is soon made opaque by atomized antimony and carbon. The proper functioning of the arc light can then be seen from the ampmeter, whose deflection should remain 10-20 percent below the maximum observed during a short circuit. If the cooling water is cold enough, the apparatus will operate for minutes without supervision. After a time, antimony globules often collect on the graphite electrode, as can be seen by removing the stopper together with the electrodes; these can be made to disappear by temporary reversal of polarity. One cm of electrode burns away in $\frac{1}{2}$ hour, so that the anode is used up in about 3 hours.

The very sharp-smelling, brownish-red liquid is drained off and filtered and, then after the remaining black ooze, which is intermixed with tiny metallic antimony globules, has been washed out with carbon disulfide, shaken for an hour with about 200 g of mercury and some phosphorous pentoxide. The filtrate is reduced in a water bath to 100 ccm and is now a pure, approximately 1-percent solution of C_3S_2, which can be stored in a dark place for weeks without decomposing. Its concentration can be exactly determined by evaporation with bromine (see above).

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*It is important that its diameter be larger than that of the anode, so that the latter will burn away evenly.

The dilute C_3S_2 solutions obtained by the von Lengyel process or by overheating carbon disulfide vapors in a silica tube can be treated in the same way. Because of their higher "residue" content, however, they should be shaken longer and with more mercury. It is recommended that the greater part of the impurities first be removed by several days contact with copper wire net.

To isolate pure carbon subsulfide, the 1-percent solution is shaken once more with a small amount of mercury and phosphorous pentoxide and reduced in the water bath to about $^1/_5$, then in the vacuum of a water-jet pump at room temperature until the carbon disulfide is removed. The remaining red liquid is distilled in a high vacuum in a well-cooled condenser. We evacuated with a Gaede mercurial air pump and cooled the condenser with industrial liquid ammonia (about -40 °C). The carbon subsulfide condenses in the condenser to an almost quantitative yield of a solid, yellowish-red substance, which melts at room temperature to a glowing red, highly refractive liquid. Produced in this way, it is of a purity attainable with few substances. This was a result especially of the exactly coinciding pressures and melting points of the various preparations. It was entirely free of carbon disulfide; for when large quantities were polymerized in closed, evacuated vessels, the pressure after which all C_3S_2 was converted was exactly zero (see below).

0.1679 g substance: 0.2214 g CO_2, 0.0025 g H_2O.
0.1272 g substance: 0.5937 g $BaSO_4$;
0.1252 g substance: 0.5854 g $BaSO_4$.
C_3S_2. Calculated: C 36.0, S 64.0, H –.
Found: C 36.0, S 64.1, 64.3, H 0.17.

Definition of molecular weight: 0.2917 g substance: 14.32 g CS_2 solution; increase in boiling point: 0.46 °C. Found: M 106.3 (Theoretical Molecular wt.: 100.1).

Definition of vapor density. We filled an approximately 200 ccm evacuated flask at 60 °C with saturated C_3S_2 vapor; melted it away from the feed apparatus with a short mercury gauge and a flask with a small tube attached; heated it together with gauge, etc. to about 70 °C; measured pressure and temperature; and subsequently determined the quantity of the C_3S_2 contained in the flask by cooling the attached tube in liquid air, so that all of the C_3S_2 in it condensed; melted off, weighed, opened, purified of C_3S_2, and weighed again empty. 197.3 ccm, 70.5 °C, 12.5 mm, 0.0124 g substance.

Found: D = 53.5 (Theoretical density: 50.0).

Density (by the suspension method in $CaCl_2$ solution) at 15°C: 1.319*.
Melting point: -0.5°C.

Vapor pressures: at	20	30	40	50	60	70	80	90°C
	1	2	4	8	13	21	32	48 mm.

Melting point and vapor pressures of preparations produced in different ways were found to be completely the same. Above 90°C the pressures could not be determined because of the tendency of the substance to decompose. With its pressure of about 1 mm, carbon subsulfide evaporates rather quickly when exposed to air at room temperature.

C_3S_2 remains unchanged when mixed with carbon disulfide and benzene. A solution in alcohol decomposes after standing for a short time. We investigated the carbon disulfide solutions more carefully. When greatly diluted they are salmon-colored; stronger concentrations are yellowish-brown. The 1-percent solution lets the red portion of the spectrum to 540 μμ and weak light from 435-415 μμ through; the 0.1-percent solution shows a wide band of absorption in the green and blue portions between 530 and 455 μμ. With a C_3S_2 content up to 1 percent, the solutions are quite stable when kept in the dark; with a higher content, they gradually precipitate black substances, polymerization products of C_3S_2. The dilute solutions do the same when exposed to sunlight. It is difficult to decompose all of the C_3S_2 in this way, because of dark color of the solution and the black crusts which form on the sides of the vessel hinder any further effect of the light. On the other hand, heating the carbon disulfide solution to 200°C brings about complete separation of everything that was dissolved. Metallic copper and mercury remain clean in carbon disulfide solutions of C_3S_2. The sulfides which are formed from both metals in solutions of sulfur in carbon disulfide extract the C_3S_2 from the carbon disulfide solutions, as already mentioned. This occurs rather quickly with copper, far more slowly with mercury. The weight of the copper sulfide, which we used to study the phenomenon more closely, increases by that of the C_3S_2 lost. Apparently, a slow addition reaction takes place between C_3S_2 and the sulfide. It is not a matter of polymerization of the C_3S_2, shortly to be discussed; for the end produce of that process is not altered by chlorine in a watery solution, while the substance formed by C_3S_2 and copper sulfide is easily oxidized with such a solution.

*The value of 1.27389 published by V. Lengyel must be based on an error. It approaches the density of carbon disulfide (1.27) so nearly, that it cannot be explained by the contamination of the C_3S_2 with CS_2.

Especially striking is a characteristic of C_3S_2 already studied by von Lengyel: that of spontaneous polymerization into carbon-like, non-volatile black substances. At room temperature pure C_3S_2 shows the beginning of this spontaneous decomposition only after a few hours, by turning dark and cloudy. At 100 °C it changes in only a few minutes into the solid black mass. Von Lengyel thought that some carbon disulfide forms in the process. With pure C_3S_2, however, this is not the case. When heated long enough in evacuated vessels, the pressure (which was at first, for example, 48 mm at 90 °C) returns gradually to zero. Even traces of carbon disulfide would make themselves noticeable through continuing pressure. The measurement of pressure serves as a very simple method of determining the quantities of C_3S_2 still present at any one time and from that its rate of decomposition. We conducted two such experiments, using vessels whose ratios of surface to volume were very different. One (Experiment I) consisted of a U-tube 4 mm wide and 4 ccm in volume; the other (II) of a globe 300 ccm in volume. The surface areas as related to the unit of volume had a ratio for I and II of 13:1. Temperature for the experiment was 90 °C. We observed the following pressures, furnished with the necessary adjustments:

I.	Time in minutes:	0	5	10	15	20	25	30	45	65	∞		
	Pressure in mm:	42.3	27.0	18.5	13.3	10.2	8.1	6.5	3.6	1.6	0.		
II.	Time in minutes:	0	5	20	35	65	95	125	215	305	425	545	985	∞
	Pressure in mm:	42.3	40.0	35.8	32.0	26.8	22.6	19.8	13.6	10.0	7.0	4.6	2.0	0.

The rates of decomposition at the beginning have ratios for I and II of close to 13:1 (decrease in pressure of $15\frac{1}{2}$ mm for I in 5 minutes, for II in 65 minutes); they are therefore proportional to the ratios of vessel surface area to unit of volume, i.e., the deciding process takes place on the sides of the vessel. The course of the decomposition is not determined by adsorption[4], as would at first be thought most probable under these circumstances. In that case, the reaction velocity would increase with the decrease in C_3S_2 concentration, while in fact it becomes smaller. The nature of the reaction corresponds far more to that of a bimolecular reaction, as the approaching constancy of the values calculated from Experiment II, using the equation

$$k = \frac{x}{ta(a - x)}$$

(x is the quantity decomposed according to the time, t) shows:

t	x	k 10^4	t	x	k 10^4
0	0		125	0.53	90
5	0.05	105	215	0.68	99
20	0.15	90	305	0.76	104
35	0.24	90	425	0.84	122
65	0.35	90	545	0.89	148
95	0.47	93			

The nature of the reaction for I lies between that of a mono- and that of a dimolecular reaction. Given the small volume of the vessel, however, the change in surface area caused by the rapid separation of the polymerization product must here have a disrupting effect.

One of the possible explanations for the course of this reaction would be in the assumption that 2 molecules of C_3S_2 first react with one another in the vapor and that the condensation product formed by them polymerizes on the side of the vessel to the solid black substance, which apparently has a high molecular weight.

This black substance is not altered by water, soda lye, hydrochloric acid, or chlorine water. Heated in a vacuum, it decomposes for the most part into carbon disulfide and, at dull red heat, a stable carbon-like residue, which still contains large quantities of sulfur (an analysis showed 39 percent). This decomposition corresponds entirely to the behavior of carbon suboxide polymerization products[5].

Carbon subsulfide reacts very easily with aniline according to the equation:

$$C_3S_2 + 2C_6H_5 \cdot NH_2 = CH_2(CS \cdot NH \cdot C_6H_5)_2$$

and yields thiomalonic anilide, produced by Reissert from malonic anilide and phosphorous pentasulfide[6].

Ten ccm of a 5-percent solution of freshly-made C_3S_2 benzene were mixed with 10.1 ccm of a 9.3-percent benzene-aniline solution — that is, with a somewhat greater quantity of aniline than that specified by the above equation. The mixture became heated, lost the sharp smell of the C_3S_2, and hardened to a paste of crystal needles*. The mother liquor gradually turned dark red. We diluted it with 40 ccm

*The yield of crystals is much lower when dilute solutions are mixed. Secondary reactions are then dominant.

of benzene and, after letting it stand in the cold for $^3/_4$ of an hour, filtered out the crystals. The amount of the light brown crystals, washed with a little benzene and dried, came to 1.1 g (calculated for thiomalonic anilide: 1.4 g). The substance was again crystallized from 60 ccm of benzene. Yield: 0.85 g of yellowish, matted needles, which melted at 150°-151°C to a red liquid and did not change their melting point after recrystallization from alcohol. Reissert gives a melting point of 149°C for thiomalonic anilide. The analysis of our substance corresponded exactly to thiomalonic anilide.

0.1539 g substance: 0.3571 g CO_2, 0.0688 g H_2O.
0.1511 g substance: 12.7 ccm N (18°C, 749 mm).
0.1070 g substance: 0.1736 g $BaSO_4$

$C_{15}H_{14}N_2S_2$. Calculated: C 62.9, H 5.0, N 9.8, S 22.4
Found: C 63.3, H 5.0, N 9.6, S 22.3 .

That our preparation is identical with that of Reissert is not to be doubted. Since the constitution of the latter is certain, a close connection between carbon subsulfide and malonic acid is thereby indicated. Therefore, C_3S_2 corresponds in its structure to carbon suboxide, which it also strikingly resembles in its unbearably sharp smell and its tendency to polymerization.

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13. ABSTRACT

Presented is a thorough study of carbon telluride, the latter of which is always mixed with a sharp smelling substance that consists of sulfur and carbon and is referred to as carbon subsulfide. For conducting the investigation of carbon telluride the authors required a more exact knowledge of the carbon subsulfide. They produced it in an entirely pure state; tested different ways of producing it; specified certain constants of the pure preparation; and were able to prove, through the smooth transition of C_3S_2 combined with aniline into the already familiar compound thiomalonic anilide, that it must be S:C:C:C:S, an anhydride of thiomalonic acid.

The authors show that C_3S_2 can be produced from carbon disulfide by various methods. Dilute carbon disulfide solutions are always obtained, in which in addition to C_3S_2, changing quantities of impurities, such as sulfur and perhaps also carbon polysulfides, are to be found. These impurities which remain behind when the solution is evaporated while C_3S_2 evaporates can be removed by treating the liquid with copper, and also by the more effective and much quicker method of shaking with mercury. The carbon subsulfide content of the remaining pure C_3S_2 solutions was determined by evaporation with excess bromine and weighing of the residue, whose composition corresponds to the formula $C_3S_2Br_6$. It is shown that the conversion of C_3S_2 into the bromide proceeds quantitatively.

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Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Carbon telluride Carbon subsulfide Carbon disulfide Metal sulfide Arsenic electrodes Molecular weight Vapor density Vapor pressure						

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